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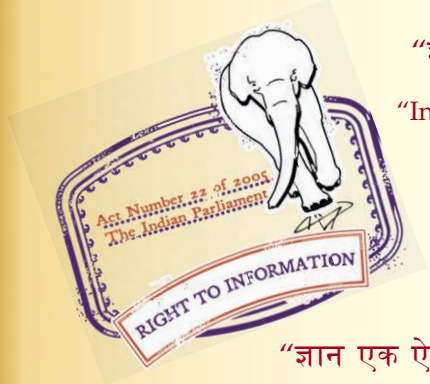

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"Knowledge is such a treasure which cannot be stolen"

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IS 10117 : 2000
(Reaffirmed 2006)

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(पहला पुनरीक्षण)

Indian Standard

CODE OF PRACTICE FOR PASSIVATION OF STAINLESS STEEL ARTICLES, INDUSTRIAL EQUIPMENTS AND COMPONENTS INCLUDING PIPELINES

(First Revision)

ICS 25.220.01, 77.140,20

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110 002

December 2000

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Corrosion Protection Sectional Committee had been approved by the Metallurgical Engineering Division Council.

The stainless steel articles including industrial hardware owe their resistance to corrosion to the formation of a protective oxide film on its surface. Passivation treatment is normally employed for enhancing the formation of the protective film on the surface, by immersion in an oxidizing acid solution. This standard has been prepared for specifying the process details for the passivation treatment and methods for testing the passivated surfaces. This pretreatment is generally applied to stainless steel surgical instruments, cutlery, tubes and various other industrial equipments and hardware.

In this revision the scope of the standard has been widened to include industrial equipments including pipelines. This revision of the standard incorporates soak cleaning and mechanical descaling as additional methods for surface preparation.The recommended passivation treatments for different stainless steels have been detailed in this version of the standard. While ferroxyl test and high humidity test have been included as tests for resistance to corrosion, the potential-time measurement has been introduced to confirm passivation of the surface.

With the above changes the specification has been made much detailed and user friendly.

Indian Standard

CODE OF PRACTICE FOR PASSIVATION OF STAINLESS STEEL ARTICLES, INDUSTRIAL EQUIPMENTS AND COMPONENTS INCLUDING PIPELINES

(First Revision)

1 SCOPE

1.1 The industrial stainless steel hardwares find a wide spread use in oil, petrochemical, drugs and pharmaceutical sector, etc. The danger of unexpected failure due to even a small abuse necessitates the industrial application of the stainless steel hardware only after a suitable passivation through proper inspection and testing.

1.2 This standard covers the processing of stainless steel industrial equipments, components including pipelines to produce a film essentially of metal oxides that are intended to be used for corrosion protection. Methods for testing the corrosion resistance of passivated industrial hardware have also been specified in the standard.

2 QUALITY OF CHEMICALS

The quality of chemicals used in the passivation treatment shall be such that it does not affect the process where the equipment is put to use.

3 SURFACE PREPARATION

3.1 Soak Cleaning

3.1.1 All surfaces shall be free from scale, oil, grease and foreign matter. Oily and greasy articles or components should be treated in soak cleaner at 70-90°C. These soak cleaners may contain hydroxide, carbonate and/or silicate along with a wetting agent and should be free from pollutants like cyanide, flouride or phosphate. Pre-cleaning will not be required when only thin oxide is present on the surface.

3.2 Pickling

3.2.1 After soak cleaning the article or component should be pickled in a solution containing 100 ml of concentrated nitric acid (r.d = 1.42) and 20 ml of hydrofluoric acid (r.d = 1.24) per litre at a temperature of 60 – 65°C in a PTFE container. This should be followed by pickling in a solution containing 100 ml of concentrated sulphuric acid (r.d = 1.84) per litre at a temperature of 80° – 90°C. This treatment shall remove the grit and scale from the surface and provide a fairly white surface. For pickling of specific types of stainless steels, reference may be made to Table 1.

3.2.2 Forgings and castings are usually not pickled but forgings can be pickled as given in inspection procedure to determine the presence of surface defects.

3.2.3 HNO_3-HF solutions may intergranularly corrode certain alloys (316, 317 and other Mo containing alloys) if these are sensitized by improper welding/ heat treatment. The crevices so formed may accumulate halides and subsequently result in stress corrosion cracking or crevice corrosion, if left under wet condition. Therefore, such alloys should not be pickled in sensitized condition and consideration should be given to stabilized/low carbon grade alloys if acid pickling after welding is unavoidable.

3.2.4 Severe pitting may result from prolonged exposure to certain acid solutions if the solution becomes depleted or if the concentration of metallic salts becomes too high as a result of prolonged use of the solution. The concentration of iron should not exceed 5 % by weight. Care should be taken to avoid over pickling.

3.3 Materials should be vigorously brushed with hot water (60-80°C) and a bristle brush or with high pressure water jet on completion of pickling. *p*H of final water rinse should be between 6 and 8 for most applications and 6.5 and 7.5 for critical applications. To minimize staining, surfaces must not be permitted to dry between successive steps of the acid descaling and rinsing procedure. Thorough drying should follow the final water rinse using demineralized water.

3.4 Hardenable 400 series alloys, maraging alloys and precipitation hardening alloys in the hardened conditions are subjected to hydrogen embrittlement or intergranular attack by acids. Wherever possible descaling by mechanical means is recommended for such alloys. If acid pickling is unavoidable parts should be heated at 120-150°C for 24 hours immediately following acid treatment to drive off the hydrogen and reduce susceptibility to embrittlement.

3.5 Mechanical Descaling

3.5.1 Mechanical descaling methods include abrasive blasting, power brushing, sanding, grinding and chipping. Surfaces to be descaled may have to be precleaned .Particular care must be taken to avoid damage by mechanical methods while descaling thin sections, polished surfaces, and close - tolerance parts. After mechanical descaling, the surfaces should be cleaned by scrubbing with hot water and fibre brushes, followed by rinsing with clean, hot water.

Grinding wheels and sanding materials should not contain iron oxide, zinc or other undesirable materials that may cause contamination of metal surface. Grinding wheels, sanding materials, and wire brushes previous used on other surfaces should not be used on stainless steel. Wire brushes should be of a stainless steel which is equally corrosion resistance to the material being worked on.

Clean, previously unused abrasives, such as glass beads or iron free silica or alumina sand are recommended for abrasive blasting. Steel shot or grit is generally not recommended because of the possibility of embedding iron particles. The use of stainless steel shot or grit reduces the danger of rusting and iron contamination, but cannot completely eliminate the possibility of embedding residues of iron-oxide scale.

4 PASSIVATION TREATMENT

4.1 The pickled component or article should be subjected to any one of the passivation treatments listed in Table 2 for formation of the passive oxide film.

4.2 Passivation of Free Machining Stainless Steel

4.2.1 The following steps should be taken while passivating non free machining as well as free machining stainless steels with the alkaline - acid-alkaline technique:

— Alkaline rinse : soak the parts for 30 minutes in 5 wt percent NaOH at 70-80°C.

— Acid rinse : Immerse the part for 30 minutes in 20 vol percent.

HNO_3 plus 2.2 g/l $Na_2Cr_2O_7.2H_2O$ at 50-60°C.

— Alkaline rinse : Immerse for 30 minutes in 5 wt percent NaOH at 70-80°C.

— Water rinse and dry: Rinse in water using demineralized water and dry using hot air to avoid formation of patches, etc.

For recommendations on passivation of various grades of precipitation hardening non free machining as well as free machining grades of stainless steels in different surface conditions reference may be made to Table 2.

4.3 Passivation of Stainless Steel Tanks

4.3.1 Stainless steel components like tanks, pipelines, etc, shall be filled with a solution containing 5 ml concentrated nitric acid (r.d = 1.42) and 5g of sodium or potassium dichromate per litre. Small parts shall be immersed in a solution containing this solution. The solution shall be held in the equipment either for a minimum of one hour at the boiling point of this solution or for any one of the following minimum periods at the temperatures indicated:

Time	*Temperature*
24 hours	25°C
15 hours	35°C
8 hours	50°C
4 hours	65°C
2 ½ hours	80°C

4.3.2 Passivation treatment shall be carried out in such a manner that the formation of the oxide coating is complete. Proper attention shall be paid to duration of passivation treatment, solution temperature and composition. The material used for construction of the passivation plant shall be such that they have no adverse effect on the quality of the passive film produced.

4.4 Rinsing and Drying

Passivated articles shall be rinsed thoroughly in cold running water to remove any residue of passivation solution. After final rinsing, the passivated articles shall be dried thoroughly by circulation of hot air. While drying, special attention should be paid to the parts, that contain pocket crevices.

4.5 Proper personal protection including face shields, rubber gloves and rubber protective clothing must be provided when handling acids and other corrosive chemicals. Adequate ventilation and strict personnel-access controls must be maintained in areas where such chemicals are being used.

5 INSPECTION AND TESTING

5.1 Visual Inspection

The passivated surface on visual examination shall be free from untreated patches and flaky and uneven deposits.

5.2 Test for Resistance to Corrosion

The resistance of the passivated articles to corrosion shall be tested by means of the following tests. The specific test shall be agreed to between the purchaser and the supplier.

5.2.1 *Copper Sulphate Test*

Copper sulphate test should not be used for martensitic and lower (<16 percent Cr) ferritic stainless steels of 400 series, for articles to be used in food processing industry/surgical/dental applications.

The test solution is prepared by first adding sulphuric acid to distilled water (CAUTION — Always add acid to cold water) and then dissolving copper sulphate in the following proportions:

Distilled water	: 250 cm^3 batch
Sulphuric acid (H_2SO_4)	: 1 cm^3
Copper sulphate ($CuSO_45H_2O$)	: 4 g

Swab the surface to be inspected with test solution, applying additional solution if needed to keep the surface wet for a period of 6 minutes the specimen shall be rinsed and dried in a manner not to remove any deposited copper. Copper deposit will indicate the presence of free iron.

5.2.2 *Ferroxyl Test*

5.2.2.1 *Solution test*

It is a highly sensitive test and should be carried out only when even traces of free iron or iron oxide might be objectionable. It can be used on stainless steel surfaces to detect iron contamination, including iron tool marks, residual iron salts from pickling solutions, iron dust, iron deposits in welds, embedded iron or iron oxide, etc. The test solution is prepared by adding nitric acid to distilled water and then adding potassium ferricyanide in the following proportions:

Distilled water	94 wt percent	1 000 cm^3
Nitric acid	3 wt percent	20 cm^3
Potassium ferricyanide	3 wt percent	30 gm

Apply the solution with aluminium, plastic, glass or rubber atomizer having no iron or steel parts or a swab.

The appearance of a blue stain (within 15 sec of application) is an evidence of surface iron contamination (several minutes may be required to detect surface iron oxide scale). The solution should be removed from the surfaces as quickly as possible after testing using water or scrubbing with a fibre brush. Flush the surface with water several times after use of vinegar or acetic acid.

5.2.3 *Boiling Water Test*

The passivated articles/components should be brushed with soap and water, rinsed in distilled water and dried. They should be boiled in distilled water for 30 minutes and left immersed for 24 hours. The articles are considered to have passed the test, if rust does not appear on more than 2 percent of its surface This may be assessed by visual examination.

5.2.4 *High Humidity Test*

Subject the surface to 95 to 100 % humidity at 38 to 46°C in a suitable humidity cabinet for 24 to 26 h. After completion of this test, the surface should show no evidence of rust stains or other corrosion products.

5.2.5 *Pitting Test*

The passivated article should be immersed in a solution containing 40g of sodium chloride and 5g of potassium ferricyanide per litre for 10 minutes, cleaned and dried. Appearance of blue spot indicates pitting, and the article is not considered to have passed the test, if pitting occurs.

6 INSPECTION AND TESTING

6.1 Potential Time Measurement

The passivated article should be immersed in solution containing 27 ml of concentrated sulphuric acid (r.d = 1.84) at room temperature and the potential of the sample should be monitored as a function of time for 10 minutes using saturated calomel electrode as reference electrode and digital multimeter. Any potential shift in the positive direction (a shift of $\geq$ +150mV) compared to the unpassivated sample shall be an indication of passivation of the surface.

Table 1 Acid Pickling of Stainless Steel

(*Clause* 3.2.1)

Alloy	Condition	Sulution, Volume Percentage	Temperature °C	Time Minutes
AISI 200,300 and 400 series, precipitation hardening and maraging alloys (except free machining alloy)	Fully annealed only	H_2SO_4, 8-11 percent	65-80	5-45
AISI 200 and 300 series; 400 series containing Cr 18 percent or more, precipitation hardening alloys, (except free machining alloys)	Fully annealed only	HNO_3, 15-25 percent plus HF, 1-8 percent	20-80	5-30
All free-machining alloys and 400 series containing less than 16% chromium	Fully annealed only	HNO_3, 10-15 percent plus HF 1-1.5 percent	20-60	5-30

NOTES

1 The table is applicable to the equivalent cast grades of wrought alloys also.

2 Shorter time duration and lower temperatures are for low alloy stainless steels.

3 After pickling and water rinse, an aqueous caustic permanganate solution containing NaOH 10 wt% and $KMnO_4$ 4 wt% at 70-80°C for 5-60 minutes may be used for final dip to remove the smut/soot followed by thorough water rinsing and drying.

Table 2 Passivation Treatment for Stainless Steels

(*Clauses* 4.1 *and* 4.2.1)

Sl No.	Grade	Surface Condition	Passivation Treatment
	A Non-Free Machining Grades		
i)	200 and 300 series , 400 series Precipitation hardening and maraging alloys with > 16 % Cr	Annealed, cold rolled or work hardened with dull or dull non reflective surfaces	20-50 Vol% HNO_3 at 50 to 70°C for 10-30 min or 20-50 % at 20 to 40°C for 30-60 min
ii)	200 and 300 series, 400 series Precipitation hardening and maraging alloys with > 16 % Cr	Annealed, cold rolled or work hardened with bright machined or polished surface	20-40 Vol% HNO_3 + 2-6 wt% $Na_2Cr_2O_7$ $2H_2O$ at 50-70°C for 30-60 min
iii)	400 series maraging and precipitation hardening alloys with < 16% Cr	Annealed or hardened with dull or non reflective surfaces	20-50 Vol% HNO_3 at 45 to 50°C for 20-30 min or at 20-40°C for 60 min
iv)	400 series maraging and precipitation hardening alloys	Annealed or hardened with bright machined or polished surface	20-25 Vol% HNO_3 + 2-6 wt% C for $Na_2Cr_2O_7$ $2H_2O$ at 50-55°C for 15-30 min or at 20-40°C for 30-60 min
	B Free Machining Grades		
i)	200, 300 and series	Annealed or hardened with bright machined or polished surfaces	20-50 Vol% HNO_3 + 2-6 wt% $Na_2Cr_2O_7$ $2H_2O$ at 25-50°C for 25-40 min or 1-2 Vol% HNO_3 + 1-5 wt% $Na_2Cr_2O_7$ $2H_2O$ at 50-70°C 10 min
ii)	400 series with > 1.25 % Mn or > 0.40% S	Annealed or hardened with bright machined or polished surfaces	40-60 Vol% HNO_3 + 2-6 wt% $Na_2Cr_2O_7$ $2H_2O$ at 50-70°C for 20-30 min

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This Indian Standard has been developed from Doc : No. MTD 24 (4217).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at : Prabhat Offset Press, New Delhi-2